

National Bureau of Standards



TECHNICAL NEWS BULLETIN

VOLUME 32

SEPTEMBER 1948

NUMBER 9

US.93 5671

TECHNOLOGY

Measurement of Radioactive Isotopes

The determination of the number of radioactive atoms in a sample of a radioactive isotope has become a matter of importance since these isotopes have not only become an article of commerce but are also used in biological and medical experiments and even in clinical treatment of diseases. Uniform quantitative results in all laboratories can be obtained only by using procedures that will yield absolute measurements or by the use of uniform standards of comparison that will, under proper conditions of measurement, give the same result whenever the determination is made.

The National Bureau of Standards has therefore issued standard samples of radioisotopes whose radioactivity has been accurately determined, and is planning to issue samples of additional radioisotopes as they become available. This follows logically the active role which the Bureau has long taken in the field of radioactive standards and measurements, and is in line with the Bureau's responsibility to furnish standards for precision measurement in all branches of the physical sciences.

Two factors are involved in the preparation and use of these samples. First, methods and standards that are correct in principle must be used. Second, a reasonable degree of accuracy must be attained in all steps of the process not only of producing but also of using the standards. In order that the biological, medical, educational, and industrial laboratories to which the standard samples of radioisotopes are issued may use them in a way that will assure correct and reliable re-

sults, comprehensive and accurate data have been made available.¹

Essentially the measurement of a sample of a radioisotope is the measurement of the number of radioactive atoms present. This determines the strength of the source and involves three basic concepts: (1) The total number of radioactive atoms present, N ; (2) the disintegration, or decay, constant, usually represented by λ , and obtained from the half-value period, T , by the relation

$$\lambda = \frac{\log_e 2}{T} = \frac{0.693}{T}$$

and (3) the disintegration rate, or "activity", λN , the number of atoms disintegrating in unit time.

Measurement of the rate of disintegration, that is, the number of atoms disintegrating in unit time, is necessary in order to determine the total number of atoms present. The disintegration rate is of course equal to the number of nuclear particles, usually beta particles or positrons, which are emitted per unit time from all atoms which disintegrate within this interval of time. This is true because, in general, one of these particles is emitted for each disintegration which takes place. Important exceptions to this general rule will be discussed later.

The requirement that all disintegration particles must be counted per unit time is by no means simple. The

¹ See National Bureau of Standards Circular 473.

principal difficulty is that these particles are emitted equally in all directions so that it is practically impossible to devise detecting equipment which will record all of them. The Geiger-Müller beta-ray end-window counter, for example, has a window through which the beta particles enter from a source placed in front of the window. The counter can record only that fraction of the particles which pass through it. This represents, from geometrical considerations alone, not more than 25 percent of the particles actually emitted. At least three other factors may distort the results. They are absorption of particles in the window; absorption of particles in the source; and backward scattering of particles by the support of the source which directs through the window some particles originally emitted in other directions. The first two factors reduce the number from the correct value and the third increases it.

Conceivably the contribution of all of these factors can be evaluated experimentally for a particular source and a definite arrangement of the apparatus. This not only requires considerable work but it must be repeated whenever changes are made in either the source or the apparatus.

Radioactive Standards

Most of the difficulties in making quantitative determinations of radioisotopes can be eliminated if standard sources of the radioisotope under measurement are available. A standard source consists of a preparation of the isotope in a form convenient for use with the detector of radiation, and for which the disintegration rate is known from previous calibration.

Obviously, to be useful as a standard, a radioisotope must have a relatively long half-life. Furthermore, of those which have a sufficiently long half-life only those which can be calibrated in absolute disintegration rates are acceptable for preparation of standards. The number of isotopes which satisfy both these requirements is at present very limited.

When the standard is prepared from the *same* isotope as that to be measured only three simple precautions are required to secure reliable results: (1) Readings must be made with the standard in the same position as that at which readings are made on the sample; (2) the sample must be uniformly distributed over approximately the same geometrical area as the standard; and (3) the sample must be supported on a layer of material identical with that supporting the standard, or at least one that produces the same back-scattering effect.

Isotopes which have short half periods and a known disintegration scheme may be measured with a beta-ray standard of some other isotope with fair accuracy if it is known that a beta ray or a positron is emitted for every disintegration, and if the maximum energy of the beta-ray spectrum of the standard is not too different from that of the beta-ray spectrum of the isotope to be measured. In comparing samples of isotopes differing from the standards all precautions outlined for the comparisons of the same isotope must be employed and the thickness of the counter window in mg/cm^2 must be known. In addition, an absorption curve for the beta



TECHNICAL NEWS BULLETIN

U. S. DEPARTMENT OF COMMERCE

CHARLES SAWYER, Secretary

NATIONAL BUREAU OF STANDARDS

E. U. Condon, Director

SEPTEMBER 1948 Issued Monthly Vol. 32, No. 9

For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Subscription price \$1.00 a year; single copy, 10 cents. Published with approval of the Bureau of the Budget, dated February 14, 1947.

Contents

	Page
Measurement of radioactive isotopes.....	101
Expansive characteristics of hydrated limes.....	104
A radio-frequency mass spectrometer.....	105
Stress-corrosion of high-strength aluminum alloys.....	108
Frictional and thermal behavior in journal bearing lubrication.....	110
NBS publications.....	111

rays from the standard and a similar absorption curve for the beta rays from the sample must be taken. This procedure consists in observing the counting rates for each when different known thicknesses of aluminum in mg/cm^2 are interposed between the source and the counter. When the data are plotted on semilogarithmic paper, with counts per second on the logarithmic *y*-axis and thickness on the linear *x*-axis, the graphs will be very nearly straight lines. Extrapolating to zero thickness gives zero absorber counting rates for both standard and unknown sample, from which the disintegration rate of the unknown sample may be computed.

Since calibrated standards supplied by the National Bureau of Standards can be expected to maintain their calibration only when handled carefully by trained personnel, it is desirable that laboratories prepare their own working standards. This applies particularly to beta-ray standards deposited on metal planchets, such as the RaD+E standards. There is another advantage in the preparation of working standards in the laboratory. This permits the primary standards in many instances to be prepared as gamma-ray standards consisting of a solution sealed in a glass ampoule from which it need never be removed for use.

An example is furnished by the Co^{60} standards now available from the National Bureau of Standards. They consist of 5 ml of solution containing a total of 1.5 rutherford (1.5 $\times 10^6$ disintegrations per second) in one series, and 0.15 rutherford in another. They may be used to calibrate unknown solutions of Co^{60} by comparing the gamma-ray activity of the unknown solution

in a similar ampoule, using a gamma-ray electroscope for the purpose. The ratio of these readings, corrected for background, provides the information for computing the strength of the unknown solution in terms of a disintegration rate.

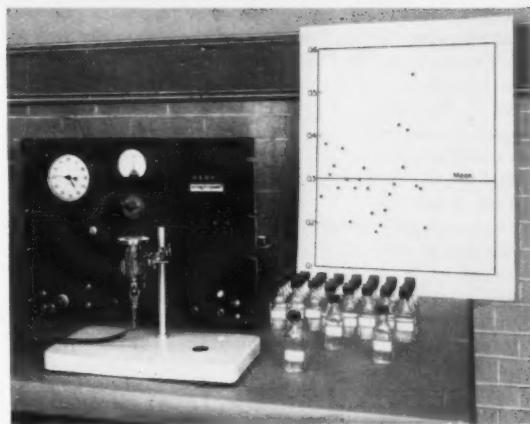
After the unknown sample has been calibrated, it can be made up to a standard volume, diluted in known ratios, and aliquots taken to produce deposits of appropriate activity for beta-ray standards.

Unknown Disintegration Schemes

Without a knowledge of the disintegration scheme, it is impossible to make reliable measurements of radioisotopes in terms of disintegration rates. The disintegration scheme serves to determine whether the mode of disintegration is such that disintegration rates can be obtained from observations which can be made conveniently. For example, if the isotope is a positron emitter but also disintegrates in part by electron capture, there is no convenient way of measuring the disintegration rate since it is difficult to determine the number of disintegrations occurring by electron capture. This capture can be detected only by virtue of the resulting characteristic X-radiation.

In the case of those isotopes for which disintegration schemes are lacking or for which it is known that the mode of disintegration does not lend itself to measurement of disintegration rates, an alternative method of comparison of activities of sources is available if the isotope emits gamma rays. This alternative does not give disintegration rates but it can, when properly applied, yield reliable comparisons of sources in various laboratories. For a particular isotope which emits gamma rays it is obvious that the intensity of the gamma radiation emitted is proportional to the amount of the isotope present. However, it is a well-known fact that electroscopes and ionization chambers used for measuring gamma rays have sensitivities which vary greatly from each other due to variations in size, materials, geometrical disposition of the source, and similar factors. Therefore, the comparison of source strengths by the gamma-ray method is a valid method only when confined to the same isotope and when using the same electroscope in the same geometrical relation to the source. This is the basic principle of all measurements of radium by the gamma-ray method.

A method has been proposed for extension of the gamma-ray method of comparison of sources to all laboratories. To obtain uniform results with such a method, no matter where the comparisons are made, a standard instrument and standard geometry are necessary. The requirement for the standard instrument is that it shall yield the same response for two equal samples of any isotope, regardless of the energies of the gamma rays emitted by these isotopes. There is a unit of gamma radiation which is defined without reference to the energy of the gamma ray. This is the roentgen, defined as "that quantity of roentgen or gamma radiation such that the associated corpuscular emission (secondary beta radiation) per 0.001293 g of air pro-



Samples of radioisotopes are compared with the NBS RaD+E beta-ray standards using a bell-type beta-ray counter (on stand) and recording instrument. In order to correlate measurements of radioisotopes in different laboratories, identical 25-ml samples of I^{131} were distributed to approximately 40 hospitals, universities, and similar institutions using this isotope or interested in its measurement. The wide range of values reported by these laboratories has been plotted on the graph at the right.

duces, in air, ions carrying 1 electrostatic unit of electricity of either sign." Therefore, an ionization instrument properly designed to measure roentgens will satisfy the requirement of a standard instrument. To determine the strength of a radioactive source the roentgens per unit must be measured at a standard distance.

These considerations have led to the suggestion² that the unit of time be one hour and the distance one meter. Convenient magnitudes are thus provided for sources in common use. Furthermore, the distance of the source from the measuring instrument is relatively large and therefore small accidental variations in this distance will not produce serious errors, because the response of the instrument varies inversely as the square of the distance from the source. With this experimental arrangement a unit is actually being used for comparison of gamma-ray sources which is defined as 1 roentgen per hour at a distance of 1 meter (roentgen per hour at a meter, abbreviated rhm).

Units

This leads to a consideration of units for expression of disintegration rates. In 1910 at the Radiology Congress in Brussels the name *curie* was applied to the amount of radon in equilibrium with one gram of pure radium. In 1930 the International Radium Standards Commission extended this definition to include the equilibrium quantity of any decay product of radium, with the comment "The Commission does not favor its extension to members outside the Ra family."³ However, the use of the curie in referring indiscriminately to

² Condon and Curtiss, Phys. Rev. **69**, 672 (1946).

³ Rev. Mod. Phys. **3**, 432 (1931).

disintegration rates has occurred in numerous places in the literature on radioactivity. As originally defined and as amended in 1930, the curie refers to the rate of disintegration of 1 gram of radium since 1 curie of radon, by virtue of its definition as the quantity in equilibrium with one gram of radium, is also that amount of radon (or any other member of the radium family), which has a rate of disintegration equal to one gram of radium.

Consequently the curie can be applied only when it is intended to refer to rate of disintegration. Although the rate of disintegration of 1 gram of radium has never been measured with great accuracy, and the exact value is in dispute to the order of 3 or 4 percent, the National Bureau of Standards has adopted the arbitrary value of 3.700×10^{10} disintegrations per second for the curie when applied to isotopes other than members of the radium family.

For many purposes a unit for the disintegration rate can be used which is smaller than the curie and which can be specified exactly and independently of any

natural constants, such as the rate of decay of radium. A convenient quantity for a unit is that quantity of radioisotope which disintegrates at the rate of a million disintegrations per second. The name *rutherford* (rd)² has been suggested for this unit. Features which make this unit simple to use include a numerical magnitude that is easy to remember, and a size that is frequently used in the laboratory. For example, a therapeutic dose of many isotopes will be of the order of 100 rd, beta-ray sources for use with mica-window, bell-type counters will be of the order of 100 to 500 μ rd, and the weakest source that can be measured with any accuracy with these counters is of the order of 1 μ rd. Tracer samples will usually be of the order of 1 rd.

The use of the rutherford in data presupposes that a disintegration rate has been measured and that this rate is expressed in terms of disintegrations per second. This procedure, if rigidly followed, removes all confusion regarding units and renders data reported from different laboratories directly comparable on an absolute basis.

Expansive Characteristics of Hydrated Limes

It is highly important that a mortar or plaster after setting in place shall not undergo any change in volume. Volume changes do occur, however, and are often the result of chemical reactions. Laboratory studies at the Bureau have led to the conclusion that a type of plaster failure characterized by the formation of bulges (large blisters) in the white coat was caused by expansion attending the slow hydration of one of the chemical components, unhydrated magnesia (MgO), of the lime used in preparing the white coat. Surveys have shown that failures are widespread and occur several years after the white coat is applied.

As a result of these studies, the Bureau was requested by other Government agencies to assist in formulating a specification that would exclude partially hydrated limes containing a high percentage of unhydrated oxides. It was proposed, therefore, that the percentage of unhydrated oxides in hydrated limes shall not exceed 3 percent by weight. This limit has recently been included in specifications of the American Society for Testing Materials and the American Standards Association.

Shortly after proposing this limit certain manufacturers of regularly hydrated dolomitic lime started producing a more completely hydrated lime. In most instances, this has been accomplished by means of large autoclaves in which the lime is hydrated at elevated temperatures and pressures.

Accelerated Test

Recognizing that chemical composition alone does not always provide an indication of the soundness of lime, the Bureau continued the investigation to develop

a suitable performance test. Since unsoundness does not usually show up for several years, an accelerated test has been proposed.

Because there is no satisfactory test procedure for ascertaining volume-change characteristics of hydrated limes themselves, it is first necessary to gage the limes with some cementitious material having hydraulic properties. Portland cement was selected as the most suitable hydraulic material for gaging. The resultant mixture is then cast into bars which, upon setting, are heated in an autoclave. The linear expansion of the autoclaved cement-lime bars thus provides a measure of the expansive properties of the lime from which they are made.

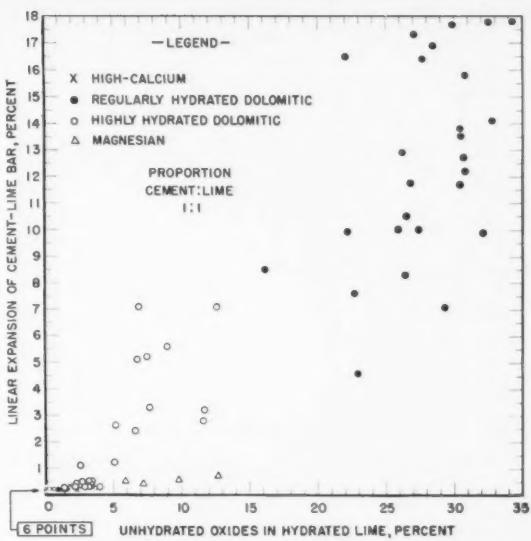


Laboratory studies at the Bureau have indicated that plaster failures of this type, which occur several years after the plaster is applied, are caused by expansion attending the slow hydration of free magnesium oxide that is present in the hydrated lime used.

Since autoclaves and auxiliary apparatus for testing the soundness of portland cement are already available in numerous testing laboratories throughout the United States, attention was directed toward developing a procedure for testing cement-lime specimens that would utilize this equipment. A specially modified autoclave was designed to measure the expansion of test specimens during the course of autoclaving.

Eighty commercial hydrated limes have been subjected to the autoclave treatment and other tests for determining the effect of variables such as the amount of lime and the use of different portland cements in the bars. On the basis of chemical analysis and percentage of unhydrated oxide, the hydrated limes were classified into four series: high-calcium, regularly hydrated dolomitic, highly hydrated dolomitic, and magnesian. In each case the relation between expansion and percentage of unhydrated oxides, the components that appear to cause the particular behavior under investigation, has been carefully determined. On the basis of the techniques developed and the data obtained, details of the test procedure have been worked out, and a limit of expansion proposed.⁴

⁴ For further technical details of this work, see Expansive characteristics of hydrated limes and the development of an autoclave test for soundness, by Lansing S. Wells, Walter F. Clarke, and Ernest M. Levin, J. Research NBS 41 (September 1948) RP 1917.



The linear expansion of autoclaved cement-lime bars provides a measure of the expansive characteristics of the limes from which they are made. Note the high expansivity of those limes containing a high percentage of unhydrated oxides.

A Radio-Frequency Mass Spectrometer

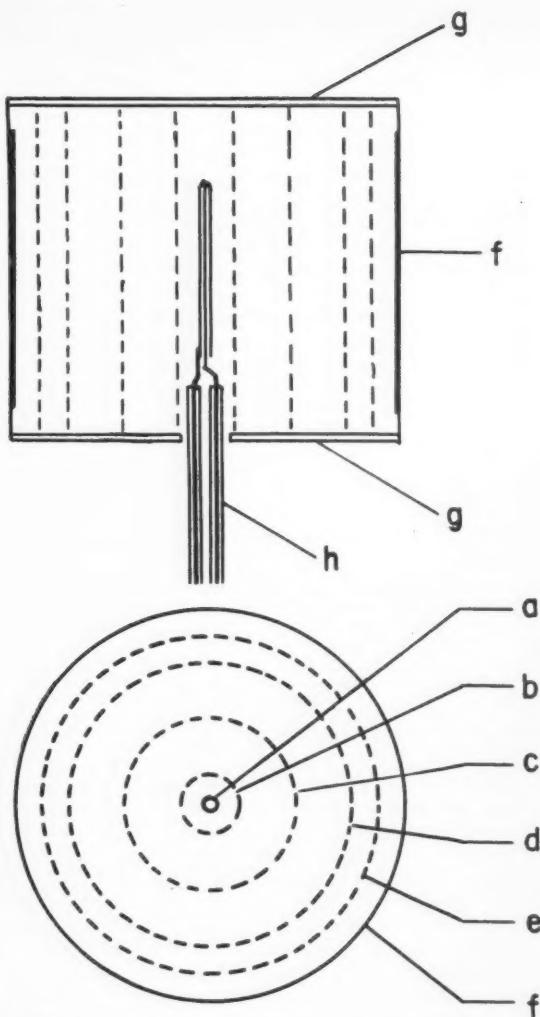
A comprehensive investigation of the conditions for the formation of negative atomic ions of the heavier metallic elements has led to the development, by Dr. Willard H. Bennett of the Bureau's physical electronics laboratory, of an extremely valuable method for detecting, separating, identifying, and measuring such ions. This is one of the most neglected fields of research in pure physics. Since negative atomic ions, consisting of atoms with extra electrons, have very low energies of formation, their study has been difficult because of the rapid dissociation of the ions in very short distances of travel; that is, before they can reach a measuring electrode. The exacting requirements of this field of research have now been met by means of a specially designed vacuum tube which provides a greatly simplified and flexible radio-frequency mass spectrometer. This spectrometer not only opens up a new means of exploring the little-known fundamentals of negative atomic ions but its special characteristics make it widely applicable for other uses, including the mass spectrometry of positive ions.

Negative atomic ions have been detected for only a few elements since they were first discovered about 40 years ago, although accepted theory has indicated the possibility of producing such ions from many of the elements. Experiments begun at the Bureau by Dr. Bennett in 1946 indicated that negative atomic ions might well exist in the many familiar forms of electrical discharge in vacuum tubes, but that they would not be detected if the distance through the tube between the

discharge and the electrode was large. It was necessary, therefore, to devise an experimental method for separating and identifying such ions within distances of only a few centimeters. This was accomplished through the new mass-spectrometer tube.

In its more advanced form, this two-stage spectrometer may be used for positive ions as well as negative ions. The equipment consists essentially of a multigrid tube in which an adjustable radio frequency is applied to two grids, while all other electrodes are held at the proper direct-current potentials and the ion current is measured at the plate. The more exacting requirements of negative-ion separation require the use of a small magnetic field produced with coils, but if positive ions are being separated, no magnetic field is needed. Because large electromagnets or tubes containing electrodes with elaborate slit systems are not required in this method, the radio-frequency mass spectrometer should find wide application where its resolution is sufficient and where the expense of the more elaborate, conventional beam-deflection equipment is not justified.

One of the principal limitations upon the resolution possible with the ordinary mass spectrometers using magnetic deflection of beams has been the spread in energies of the ions at the ion source. The percentage spread occasioned by this factor can be reduced by increasing the voltage applied to the ions before they are magnetically resolved. The extent to which this can be done is limited, however, by the magnetic field that can be obtained in a space sufficient to contain the tube.



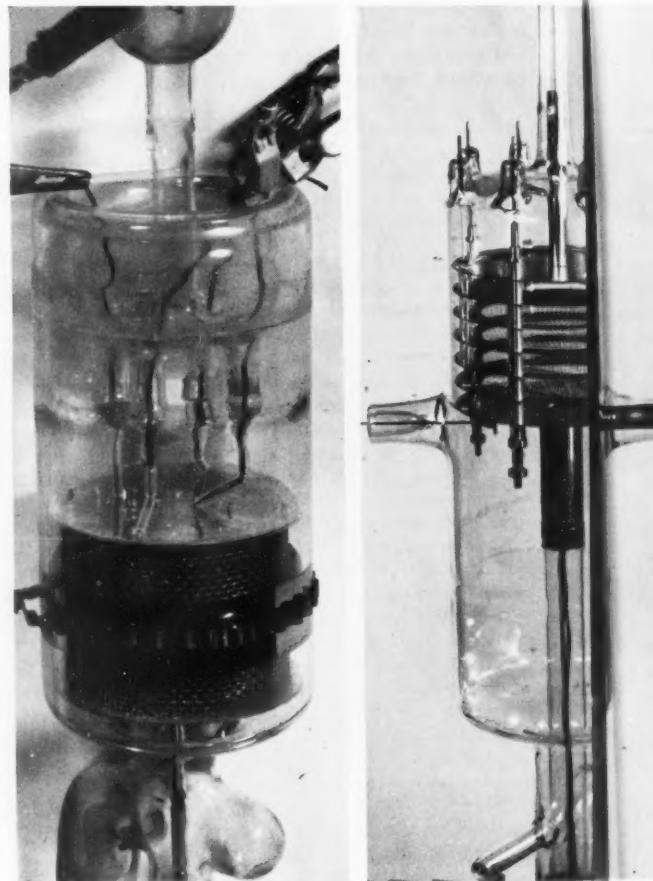
Elements of the early single-stage tube (right) include a cathode, *a*, about which are arranged four coaxial cylindrical grids (*b*, *c*, *d*, *e*) and an ion-collecting plate, *f*. The electrodes are closed at the ends with mica disks (*g*, *g*) except for the hole in the lower disk through which the glass-insulated cathode lead, *h*, extends.

In the radio-frequency mass spectrometer, this difficulty is eliminated, and the voltage of the ions can easily be pushed up at least an additional order of magnitude to any value for which insulation can be provided. The frequencies required are then increased by an amount equal to the square root of the factor by which the voltage is increased. Raising the voltage from 100 to 10,000 volts, for example, increases the frequencies tenfold, and reduces the percentage spread of mass line due to velocity spread in the ion sources by a factor of one one-hundredth.

The first successful radio-frequency mass spec-

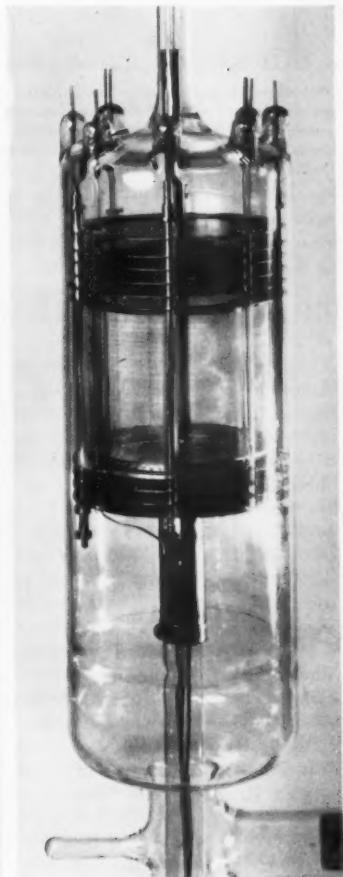
trometer, a single-stage six-electrode tube, consists of a cathode about which are arranged four coaxial cylindrical grids and an ion-collecting plate. Helmholtz coils surrounding the tube provide a 100-gauss magnetic field in a direction parallel with the axis of the tube. Such a field is required in negative-ion work to confine the electrons to the space inside the first grid, and to prevent the formation of positive ions in parts of the tube where neutralization of the negative ions may occur before they reach the detecting electrode.

In the operation of the single-stage negative-ion spectrometer tube, ions arising at the cathode are accelerated through the first grid at a velocity corresponding to the d-c potential of that grid (say 15 volts). They are further accelerated by the field due to the potential on the second grid, which is an alternating radio frequency (say 5 volts) plus a d-c potential (reduced to 10 volts). The third grid is held at a d-c potential (reduced to 5

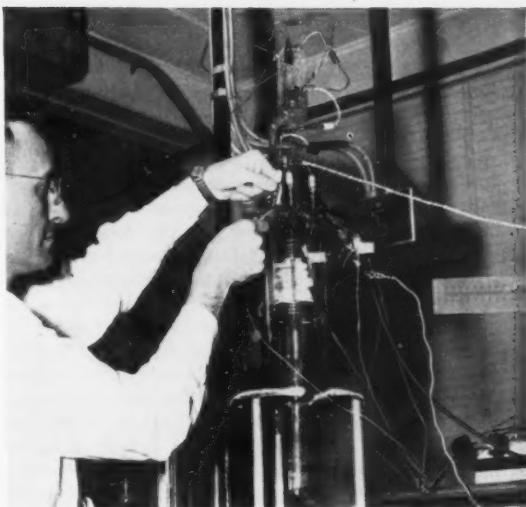


The first successful form of the radio-frequency mass spectrometer (left) of a cathode, four coaxial cylindrical grids, and an ion-collecting plate. A modified design (center) includes two stages. The use of two stages (right) considerably improves the range of readily producible radio frequencies more than the first form.

volts). Those ions which pass the first grid at the proper phase of the r-f field, and which have a mass and related velocity such that they pass through the second grid while the r-f potential changes phase, receive an additional acceleration due to the r-f field while travelling to the third grid. It was observed experimentally, verifying theoretical calculation, that ions passing the first grid at 45° $27.5'$ of phase angle of the alternating current on the second grid, and having just the right mass (and corresponding velocity) to pass the second grid at 130° of phase angle of the current on that grid, will pass each of the first three grids with the same velocity (corresponding to 15 volts), although the d-c potentials on those grids are successively reduced. This is caused by the ion picking up energy from the field of the alternating current on the second grid, as the change in phase occurs when the ion passes through this grid.



Spec^{tr} (left) comprises a single-stage six-electrode tube consisting of a collector. Helmholtz coils surrounding the tube provide a magnetic field. The center tube meets the exacting requirements of negative-ion investigation. The mass resolution which can be obtained with this kind of tube is greater than the full mass range of all the elements.



Special characteristics of a simplified and flexible radio-frequency mass spectrometer (center) developed at the Bureau make it applicable for obtaining basic data on negative atomic ions as well as for the mass spectrometry of positive ions.

A blocking potential difference, nearly equal to the maximum energy that an ion with the proper mass (corresponding to the frequency) could pick up from the alternating current, is applied between the cathode and the fourth grid. The blocking potential may be, for example, 9.6 volts negative with respect to the cathode. The collecting electrode, or ion-collection plate, is held at 40 volts positive with respect to the cathode to insure that no positive ions will reach the anode. An electrometer tube circuit and galvanometer are used for measuring the selected ion currents to the anode.

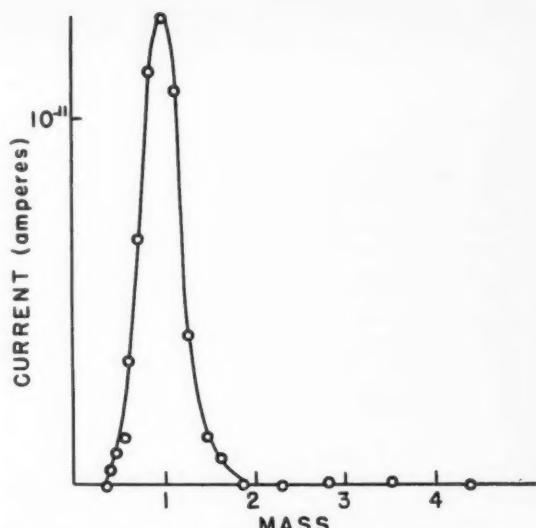
By observing the negative atomic ions produced at an oxide-coated cathode in a water-vapor atmosphere at less than 10^{-6} mm of mercury pressure, typical mass-resolution data may be obtained for plotting resolution curves. The data are taken while holding all potentials constant and varying only the applied radio frequency. For example, an a-c potential of 5 volts may be used while d-c potentials of less than 100 volts are applied to the other electrodes. The required frequency range extends from 3,300 kilocycles for a mass value of $1/4$, to 104 kilocycles for mass value 250. This range of readily producible radio frequencies more than covers the full mass range of all elements.

By extending the method to two stages in a nine-electrode tube, either with cylindrical or parallel-plane electrodes, and applying the radio frequency to two of the grids, a high order of mass resolution is obtained. Such a large number of grids would not be practical in a tube if the usual woven-wire screens were used. There is available now, however, a knitted-wire fabric that has more than 95 percent open area and with which this kind of tube can readily be constructed. This apparatus may be used in the mass spectrometry of positive ions

and in ionization studies, as well as in the present negative-ion investigations. The magnetic field is again applied in a direction parallel with the planes of the grids for negative-ion investigations. A collimating electrode containing the cathode is used to direct the ions in a direction approximately normal to the planes of the grids.

In the positive-ion mass-spectrometer tube, the ions, accelerated toward the second grid, are resolved by the application of a radio frequency on the third grid, just as in the single-stage tube. The distance between the fourth and fifth grids is selected to assure that those ions which acquire the maximum energy from the alternating-current fields at the first stage (grids 2, 3, & 4) will travel from grid 3 to grid 6 in the time that the radio frequency takes to traverse an exactly integral number of cycles. The same radio frequency is applied to the sixth grid as to the third grid and the other potentials on the second stage (grids 5, 6, & 7) are selected in a similar manner to those for the first stage. The blocking potential is applied to the anode, and the selected ions are collected there.

The simplicity and low cost of the radio-frequency mass spectrometer should make it attractive not only in those applications in which its special characteristics make it superior to any other kind of mass spectrometer, but also in those laboratories where the expense of other types of equipment is prohibitive. Though the possibilities of the new apparatus have not yet been adequately explored, preliminary experimentation at



By observing the negative atomic ions produced at an oxide-coated cathode in a water-vapor atmosphere at less than 10^{-6} mm of mercury pressure, typical mass-resolution data are obtained for plotting resolution curves.

the National Bureau of Standards indicates that many specialized uses for this kind of tube will come to light as it becomes better known.

Stress-Corrosion of High-Strength Aluminum Alloys

The current demand for larger, heavier, and faster aircraft has resulted in the development of a number of new high-strength aluminum alloys. As these materials have become available, their resistance to corrosion has been determined by the National Bureau of Standards, usually at the request of the Armed Services. In connection with the program, several aluminum alloys introduced during the war, namely, the 75S-T, R301-T, R303-T, and the artificially aged 24S-T, were studied to determine their ability to withstand the highly corrosive conditions to which military planes are subjected in tropical areas.

In the course of this investigation, methods and apparatus for accelerated laboratory tests of stressed samples in corrosive solutions were developed and applied⁵ by Hugh L. Logan and Harold Hessing of the Bureau's metallurgical laboratories. Marine-atmosphere exposure tests of the same materials under similar experimental conditions indicate a high degree of correlation between results of the laboratory tests and what may be expected in actual service.

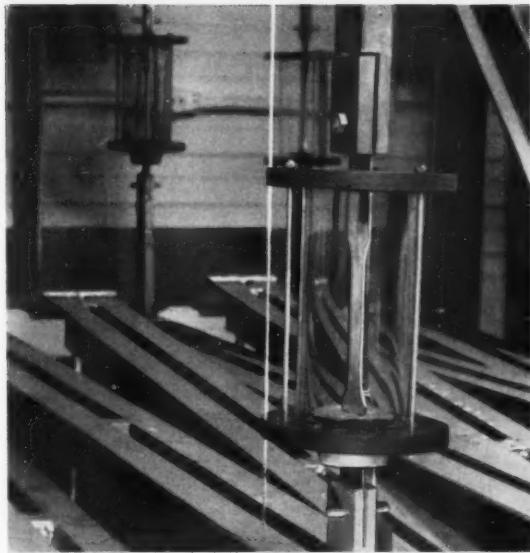
The duralumin type of aluminum-copper-magnesium alloy known as 24S-T was introduced in 1932 and within a decade had largely replaced other aluminum alloys as sheet material for aircraft construction.

Although its resistance to corrosion was generally satisfactory, under some conditions it was appreciably attacked upon exposure to a marine atmosphere or sea water. To increase corrosion resistance, it has been used in the form of a "clad" alloy, in which a duralumin sheet is sandwiched between, and integrally bonded to, two thin protective layers of commercially pure aluminum. This, however, results in some sacrifice of tensile strength. As this material has been found to be adequately resistant to corrosion in service, it was used as a standard of comparison in the marine-atmosphere testing of the new alloys.

With the outbreak of the war in 1939, even stronger aluminum alloys became desirable for aircraft use. Efforts were therefore made to develop alloys of greater tensile strength with adequate corrosion resistance. In the R301-T alloy, this result was sought by covering a duralumin-type alloy with an alloy-cladding layer of higher strength than commercially pure aluminum. In the 75S-T and R303-T alloys, tensile properties were improved by the addition of appreciable amounts of zinc. Use was also made of the discovery that aging of the commercial flat 24S-T alloy at an elevated temperature results in a marked increase in yield strength.

In the Bureau's investigation of the stress-corrosion resistance of these new materials, standard ASTM flat tensile specimens with $1/2$ -inch reduced sections were tested both in the laboratory and in a marine atmos-

⁵ For further technical details, see Stress-corrosion tests on high-strength aluminum alloy sheet by Hugh L. Logan and Harold Hessing, J. Research NBS 41, 69 (1948) RP1905.



In accelerated tests of the corrosion resistance of stressed aluminum-alloy sheet, elongated specimens of the material, supported vertically within glass cells containing a corrosive sodium chloride-hydrogen peroxide solution, are stressed by means of weights attached to levers below.

phere under a stress equal to three-fourths of the yield strength. In the laboratory, stressed samples of all alloys were continuously immersed in a sodium chloride-hydrogen peroxide solution (NaCl, 57g; 30-percent H_2O_2 , 10 ml; H_2O , 990 ml). Those containing zinc as an alloying element (R303-T and 75S-T) were also exposed in a boiling 6-percent solution of sodium chloride. Unstressed specimens were subjected to the same corrosive conditions in order that the effect of stress in increasing corrosion damage could be evaluated. All clad materials were tested with the cladding intact since the purpose of the tests was to determine the resistance of the commercial alloy as actually used rather than that of the core material itself. Losses in ultimate tensile strength and percent elongation were taken as criteria of corrosion damage.

Specimens supported vertically and stressed by means of weighted levers were tested in the sodium chloride-hydrogen peroxide solution in cells made of 60-mm Pyrex cylindrical tubes fitted at each end into slotted bakelite disks. Rubber gaskets placed between the bakelite and the glass, and rubber stoppers molded with rectangular slots slightly smaller than the grip ends of the specimens, completed the cell assemblies. Samples up to 0.064 inch in thickness were kept in the sodium chloride-hydrogen peroxide solution for 24 hours. One-eighth-inch specimens were immersed for 72 hours, the solution being renewed at the end of each 24-hour period. Breaking of a specimen under stress automatically opened a knife switch in the circuit of a solenoid counter actuated once every 6 minutes by a clock. The time required for failure of the specimen

was thus recorded in units of $\frac{1}{10}$ hour. After removal from the solution and cleaning, the specimens were broken in a hydraulic-type tensile testing machine to determine the tensile properties of the corroded material. Metallographic examinations were also made to determine the types of corrosion that had developed. For marine-atmosphere exposure tests, the specimens were supported and stressed in a similar fashion but were left exposed to the atmosphere.

Specimens immersed in boiling sodium chloride solution were stressed by bowing. This was accomplished by a device consisting of a threaded monel rod, insulating washers slotted to hold the ends of the specimen, and two nuts adjacent to the washers. Tension on the bowed specimens was controlled by adjustment of the nuts. The specimens were then placed in wide-mouthed flasks connected to reflux condensers and containing the sodium chloride solution. They remained in the boiling solution for 14 days unless earlier failure occurred.

One of the difficulties in stressing specimens by bowing has been the fact that extremely small changes in the length of the chord connecting the ends of the specimen will make appreciable changes in the stress in the outer fiber. An instrument was therefore constructed by means of which the distance from the chord to the outer fiber could be measured to 0.0001 inch and the stress computed much more precisely than by earlier methods. Essentially, this device consists of two fixed pins and, between them, a third movable pin, which is attached to the plunger of a dial gage. Stresses computed from dial-gage readings differed from stresses obtained from wire strain gages (attached to the concave and convex surfaces of the specimen) by less than 2 percent at three-fourths of the yield strength.

The results of the investigation indicate that flat, bare 24S-T aluminum-alloy sheet aged 4 hours or longer at 375° F is not susceptible to stress-corrosion cracking in either the laboratory or marine atmosphere tests, and is at least as resistant to the combined action of stress and corrosion as the commercially heat-treated



Stress-corrosion resistance of aluminum-alloy sheet is studied under actual exposure to a marine atmosphere in apparatus designed and constructed by the Bureau at Hampton Roads, Va.

but unaged material. It was found that aging a sample of this alloy for 3 hours at 385° F produced an increase in yield strength of about 25 percent above an initial value of approximately 50,000 lb per sq in., an increase in tensile strength of about 3 percent above an initial value of approximately 70,000 lb per sq in., and a decrease of about two-thirds in the initial elongation of 17 to 18 percent. Similar results were obtained when the material was aged for 20 hours at 350° F, 5 hours at 375° F, or 1½ hours at 400° F.

The samples of the other alloys that were tested, with the exception of R301-T, were found adequately resistant to stress-corrosion cracking. In general, it was apparent that the short-time laboratory tests developed by the National Bureau of Standards give a good indication of the corrosion resistance to be expected of the unclad alloys in a marine atmosphere. However, results of continuous immersion tests on clad material in the laboratory do not necessarily agree with those obtained by exposure in a marine atmosphere.

Frictional and Thermal Behavior in Journal Bearing Lubrication

The operation of a journal bearing under a steady state of temperature distribution with forced-feed lubrication is dependent upon a number of factors related both to the conditions of operation and to the geometry of the bearing. The relation of these factors to the performance of the bearing is very complex, involving not only the rate at which the heat is generated in the bearing but also the rate at which it is carried away. In an extensive investigation of these factors, carried out at the Bureau in cooperation with the National Advisory Committee for Aeronautics,⁶ a method was developed for studying bearing performance through measurements of the combined frictional and thermal behavior in journal-bearing lubrication. A four-bearing friction machine was used for this purpose. The investigation also led to a practical graphical procedure for estimating safe bearing loads.

The work included operation of bearings of two diameters, in two length-diameter ratios and three clearance-diameter ratios, and at two journal speeds. Three oils, of different viscosity, were used at three oil-inlet temperatures. Particular attention was given to the resultant effects of the combined hydrodynamic and thermodynamic actions involved in bearing operation with forced-feed lubrication.

The four-bearing friction machine, developed and used in former investigations at the Bureau, consists essentially of four test bearings on a common shaft. The bearings are mounted in self-aligning ball-bearing swivels which are prevented from rotating by flat springs. The two outer bearings are fastened to the housing while the two inner ones are located in plates that slide in guides. The complete housing floats on the horizontal shaft and acts as a cradle dynamometer. Load is applied by hydraulic jacks under the two inner bearings.

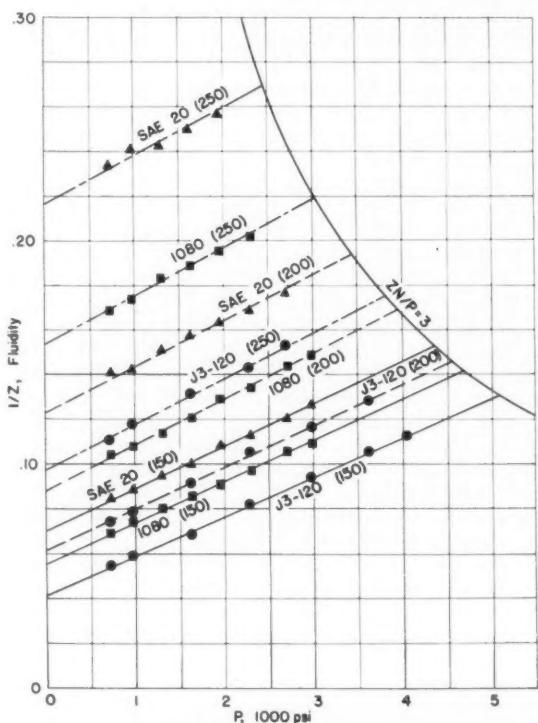
Oil is fed to the bearings through the drilled shaft

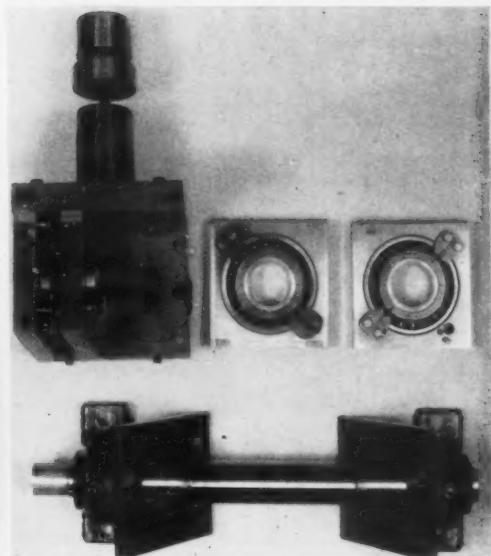
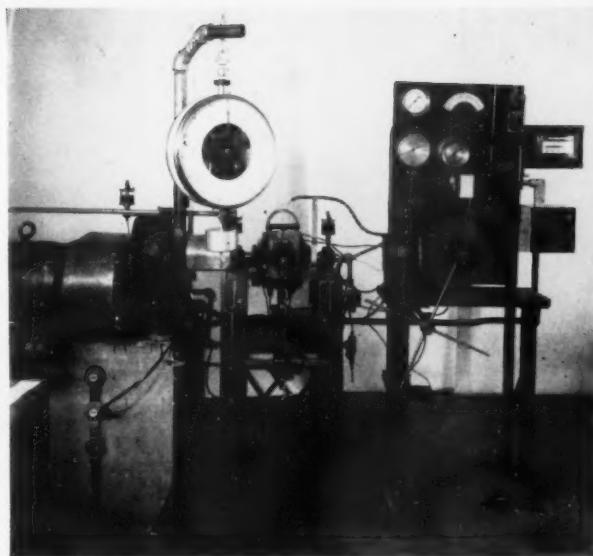
⁶ For further technical details of this work, see Measurements of combined frictional and thermal behavior in journal-bearing lubrication, by S. A. McKee, H. S. White, and J. F. Swindells. Trans. ASME 70, 409 (1948).

The straight-line relationship between fluidity and pressure provides a graphical means for estimating safe bearing loads with various oils. The intersection of each fluidity curve with the limiting curve of the generalized operating variable, ZN/P (see text), gives the maximum safe operating load. Oil-inlet temperatures in parentheses.

which has two oil holes at the longitudinal center of each bearing. A motor-driven pump and a relief valve maintain the oil-feed pressure. Bearing temperatures are measured by thermocouples embedded in the metal at the loaded sides of the bearings.

A study of the frictional-torque data for operation under load indicates that when a bearing is operating at a constant speed, using a given oil at constant oil-inlet temperature and oil-feed pressure, an increase in load produces an approximately proportional increase in frictional torque. The increase in torque with unit increase in load is influenced chiefly by the viscosity of the oil, the oil-inlet temperature, the oil-feed pressure, and the clearance-diameter and length-diameter ratios.





With the four-bearing friction machine (left) developed at the Bureau basic data have been obtained on the effects of operational and design factors upon the friction, load-carrying capacity, and operating temperature of journal bearings. The machine consists essentially of four test bearings on a common shaft, mounted in self-aligning ball-bearing swivels (right center) which are prevented from rotating by flat springs. The two outer bearings are fastened to the housing while the two inner ones are located in plates that slide in guides (lower right). The entire housing floats on the horizontal shaft and acts as a cradle dynamometer.

The results also indicate that, under these operating conditions, an increase in load causes a proportional increase in the fluidity (reciprocal of viscosity) of the oil in the bearing. For a given oil, oil-inlet temperature, and oil-feed pressure, this increase in fluidity is a function of the speed of the journal, the diameter of the journal, the length-diameter ratio, and the clearance-diameter ratio. Empirical equations have been developed to define this relationship.

The straight-line relationship between fluidity and pressure provides a graphical means for estimating safe loads. The limit for safe operation is based upon a minimum value of a generalized operating variable, ZN/P , where Z is the absolute viscosity of the lubricant, N is the speed of the journal, and P is the pressure on the bearing. The intersection of a line representing a limiting value of this variable with the straight line drawn through the fluidity-pressure data for a given set of conditions gives directly the safe bearing pressure for these conditions. This is illustrated by the accompanying graph.

In analyzing these results it is emphasized that the thermal behavior of a journal bearing operating with

forced-feed lubrication is dependent upon a complex relation that involves heat losses by radiation, conduction, and convection, as well as the heat carried away by the oil leaving the bearing. Therefore, the numerical values obtained in any series of tests are dependent upon the heat-dissipation characteristics of the specific apparatus used. In the course of the bearing-research program, however, the operating conditions were such that it was possible to differentiate between the heat carried away by the oil and the heat losses through the apparatus itself.

These data show that, under conditions typical of normal high-speed and high-temperature operation, practically all of the heat generated in the bearing is carried away by the oil. Accordingly, the straight-line relationship between fluidity and pressure should hold reasonably well in most bearing applications. In these cases considerable information on bearing performance may be obtained by making a few observations of bearing temperature for various known operating conditions. This is of considerable significance because in many bearing installations, friction measurements are not practicable.

NBS Publications

Periodicals⁷

Journal of Research of the National Bureau of Standards, volume 41, number 2, August 1948. (RP1906 to RP1914 inclusive.)

Technical News Bulletin, volume 32, number 8, August 1948. 10 cents.

CRPL-D48. Basic Radio Propagation Predictions for November 1948. Three months in advance. Issued August 1948. 10 cents.

Nonperiodicals

RESEARCH PAPERS^{7,8}

RP1896. Dipole moments and molecular association of some picrates of primary, secondary, and tertiary amines in benzene and dioxane. Arthur A. Maryott. 10 cents.

RP1897. Acid-base equilibrium constant and dipole moment of tribenzylammonium picrate in benzene from measurements of dielectric constant. Arthur A. Maryott. 10 cents.

RP1898. Amino derivatives of mannuronic acid. Harriet L. Frush and Horace S. Isbell. 10 cents.

RP1899. Properties of barium-magnesium titanate dielectrics. George R. Shelton, Ansel S. Creamer, and Elmer N. Bunting. 10 cents.

RP1900. Acid-base reactions in organic solvents. Behavior of some halogenated derivatives of phenolsulfonphthalein with different classes of organic bases in benzene. Marion Maclean Davis, Priscilla J. Schuhmann, and Mary Ellen Lovelace. 10 cents.

RP1901. Concentration of copper 63 by the countercurrent electromigration method. Samuel L. Madorsky and Sidney Straus. 10 cents.

RP1902. An instrument for the rapid production of a decimal series of potentials and its application to ballistic measurements. Howard S. Roberts and Harvey L. Curtis. 10 cents.

RP1903. Viscoelastic properties of polymer solutions. John D. Ferry. 10 cents.

RP1904. Density and refractive indices of lactose solutions. Emma J. McDonald and Anne L. Turcotte. 10 cents.

RP1905. Stress-corrosion tests on high-strength aluminum alloy sheet. Hugh L. Logan and Harold Hessing. 15 cents.

BUILDING MATERIALS AND STRUCTURES REPORTS⁷

BMS109. Strength of houses. Applications of engineering principles to structural design. H. L. Whittemore, J. B. Cotter, A. H. Stang, and V. B. Phelan. \$1.50.

COMMERCIAL STANDARDS⁷

CS73-48. Old growth Douglas fir, Sitka spruce, and Western hemlock standard stock doors. (Fourth edition. Supersedes CS73-45.) 15 cents.

TECHNOLOGIC PAPERS⁷

[REISSUE]

T170. Pyrometric practice. P. D. Foote, C. O. Fairchild, and T. R. Harrison. (February 16, 1921.) (Reprinted to meet popular demand.) 75 cents.

LETTER CIRCULARS⁹

✓LC908. List of published material relating to home building and maintenance. (Supersedes LC885.)

✓LC909. Structural clay products, stone, and masonry: Publications by the staff of the National Bureau of Standards together with a list of Federal Specifications. (Supersedes LC824.)

✓LC910. Engineering mechanics: Publications by the staff of the National Bureau of Standards. (Supersedes LC683.)

Articles by Bureau Staff Members in Outside Publications¹⁰

Prevention of dampness in basements. C. C. Fishburn. *J. Am. Concrete Inst.* (7400 Second Boulevard, Detroit 2, Mich.) **19**, 421 (1948).

A new approach to apparel sizing problems. Mansfield Lonie. *Journal of Retailing* (New York University, School of Retailing, 100 Washington Square East, New York 3, N. Y.) **23**, No. 4 (1947).

Influence of boron on some properties of experimental and commercial steels. T. G. Digges and F. M. Reinhart. *Steel Processing* (108 Smithfield Street, Pittsburgh 30, Pa.) **34**, 144 (1948).

Influence of strain aging on the fracture stress of a low carbon steel. D. J. McAdam, Jr., G. W. Geil, D. H. Woodward, and W. D. Jenkins. *Metals Technology* (American Institute of Mining and Metallurgical Engineers, 29 West 39th Street, New York 18, N. Y.) **15**, TP-2318 (1948).

Flow, fracture, and ductility of metals. D. J. McAdam, Jr., G. W. Geil and F. J. Cromwell. *Metals Technology* **15**, TP-2296 (1948).

Substituted styrenes modify polymer properties. Frank W. Reinhart. *Chemical Industries* (522 Fifth Avenue, New York, N. Y.) **62**, 235 (1948).

Penetration of leather by water under dynamic conditions. C. E. Weir, J. Carter, S. B. Newman, and J. R. Kanagy. *J. Am. Leather Chemists Assoc.* (Sec., Dept. Leather Research, Univ. of Cincinnati, Cincinnati 21, Ohio) **43**, 69 (1948).

Development of a fungicidal dressing for leather. Joseph R. Kanagy, Arbelia M. Charles, and Edward Abrams. *J. Am. Leather Chemists Assoc.* **43**, 14 (1948).

Textile testing in Germany, Part II: Serviceability evaluation of textiles. Herbert F. Schiefer, Lyman Fourt, and Richard T. Kropf. *Textile Research Journal* (10 East 40th Street, New York 16, N. Y.) **18**, 18 (1948).

Textile testing in Germany, Part III: Testing flexural and compressional properties; Part IV: Miscellaneous. Herbert F. Schiefer, Lyman Fourt, and Richard T. Kropf. *Textile Research Journal* **18**, 89 (1948).

Measurement and elimination of inertia effects in textile testing equipment by means of electric strain gages. Herbert F. Schiefer. *ASTM Bulletin* (1916 Race Street, Philadelphia 3, Pa.) No. 150, 43 (1948).

Measurement of water in gases by electrical conduction in a film of hygroscopic material, and the use of pressure changes in calibration. E. R. Weaver and Ralph Riley. *Analytical Chemistry* (1155 Sixteenth Street NW, Washington 6, D. C.) **20**, 216 (1947).

Test trail leads to flameproof fabric. Marjorie W. Sandholzer. *Textile Scope Edition*, *Daily News Record* (8 East 13th Street, New York 3, N. Y.) Sec. 3, No. 22, 20 (Jan. 27, 1948).

The effect of substituents upon the rate of fading of some sulfonphthalein indicators in alkaline solutions. Elizabeth E. Sager, Arthur A. Maryott, and Marjorie R. Schooley. *J. Am. Chem. Soc.* (1155 Sixteenth Street NW, Washington 6, D. C.) **70**, 732 (1948).

A cavity pressure method for determining the gain of hearing aids. E. L. R. Corliss and G. S. Cook. *J. Acoustical Society of America* (57 East Fifty-fifth Street, New York 22, N. Y.) **20**, 131 (1948).

New radiation meter. Henry P. Kalmus and George O. Striker. *Rev. Sci. Inst.* (57 East Fifty-fifth Street, New York 22, N. Y.) **19**, No. 2, 79 (1948).

Chromated protein films for metal protection. A. Brenner, G. Riddell, and R. Seegmiller. *J. Electrochemical Soc.* (Electrochemical Society, Inc., Mount Royal & Guilford Avenues, Baltimore 2, Md.) **93**, 55 (1948).

Physical properties of electrodeposited metals. Nickel literature survey. A. Brenner and C. W. Jennings. *Plating* (American Electroplaters' Review, P. O. Box 168, Jenkintown, Pa.) **35**, 52 (1948).

Magnetic measurement of the thickness of composite copper and nickel coatings on steel. A. Brenner and E. Kellogg. *Plating* **35**, 242 (1948).

The physical properties of electrodeposits (Progress report). A. Brenner and C. W. Jennings. *Annual Proc. Am. Electroplaters' Soc.* (P. O. Box 168, Jenkintown, Pa.) 25 (1947).

⁷ Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: *Journal of Research*, \$4.50 (foreign \$5.50); *Technical News Bulletin*, \$1 (foreign \$1.35); *Basic Radio Propagation Predictions*, \$1 (foreign \$1.25). Single copy prices of publications are indicated in the lists.

⁸ Reprints from July *Journal of Research*.

⁹ Letter Circulars are prepared to answer specific inquiries addressed to the National Bureau of Standards, and are sent only on request to persons having a definite need for the information. The Bureau cannot undertake to supply lists or complete sets of Letter Circulars or send copies automatically as issued.

¹⁰ These publications are not available from the Government. Requests should be sent direct to the publishers.

oon
and
of
street,

Jr.,
15,

W.
New
ons.
ogy.
Re-
69

R.
Am.

on of
l T.
New

om-
F.
Re-

ing
F.
a 3,

on a
anges
tical
(C.)

izer.
street,

sul-
E.
Am.
(C.)

ring
iety
20,

ker.
Y.)

, G.
etro-
ues,

era-
ting
own,

and
ting

ort).
etro-

ndent
nnal
News
oreign

o the
aving
upply
ned.
quests